A laboratory-based tender X-ray monochromator for X-ray absorption fine structure measurements*

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This paper describes the design and performance of a laboratory-based tender X-ray monochromator for X-ray absorption spectroscopy. The system enables effective absorption spectra measurement within the 2.0-9.0 keV range using Rowland circle geometry: it covers the K edge of 3d transition metals, the L edge of lanthanides and the M edge of actinides. The spectrometer is configured with a 500 mm diameter Rowland circle and integrates a 250 W liquid metal-jet X-ray source, a spherical bent crystal analyzer, and an energy-resolving silicon drift detector. The X-ray source is installed outside the vacuum chamber and remains fixed, while the analyzer crystals and detector are adjusted to change the Bragg angle while maintaining the Rowland condition. The energy resolution is $0.36 - 1.30 \, \text{eV}$ at $2.0 - 9.0 \, \text{keV}$, and the monochromatic flux is approximately $5 \times 10^5 \, \text{counts/s}$ at $7040 \, \text{eV}$. This paper highlights the main characteristics of the spectrometer and demonstrates its capabilities through selected experimental examples. The successful development of this spectrometer especially facilitates research on actinide elements, which are often constrained in synchrotron radiation experiments due to their radioactivity, thus fostering advancements in related nuclear energy fields.

Keywords: Laboratory X-ray spectroscopy, XANES, EXAFS, Tender X-ray

I. INTRODUCTION

X-ray Absorption Fine Structure (XAFS) Spectroscopy can 3 acquire local structural information, making it widely used in 4 science research [1, 2], life sciences [3], environmental stud-5 ies [4–7], and more. The advent of synchrotron radiation in 6 the 1970s significantly advanced the development of XAFS 7 technology, allowing it to evolve into a distinct experimental 8 technique integrated with synchrotron facilities. [8, 9] How-9 ever, the application process of synchrotron beam, which are 10 critical to understanding the chemistry and local structure of 11 new materials, faces challenges due to their time-consuming 12 nature. At the same time, the equipment and transportation of 13 radioactive samples for synchrotron radiation in situ XAFS 14 experiments are very complicated. Therefore, there is an ur-15 gent need to develop X-ray absorption spectrometers based 16 on laboratory scenarios to be compatible and complementary XAFS experimental conditions. 17 to

X-ray energies and instruments can be divided into soft, tender, and hard X-ray ranges [10, 11]. Currently, most laboratory spectrometers are hard X-ray absorption spectromters. These spectrometers require crystal monochromators and operate with samples in the air, so absorption is attenuated. However, the absorption and scattering of X-ray by air decreases significantly as the energy of the X-rays increases.

For example, in the actinide field, although the M-absorption edge (3.3-4 keV) of the actinide element has a smaller energy broadening than the L-absorption edge, making it more sensitive to the valence state [12], the X-ray absorption of air in this energy range is significant, and a satisfactory XAFS map cannot be obtained. This range requires a fully in-vacuum, focusing crystal spectrometer.

Commonly used XAFS spectrometers based on laboratory 33 X-ray sources have either dispersive or scanning geometry. 34 The spectrometer geometry and the diffraction characteris-35 tics of the analyzer crystal have an impact on the choice and 36 capture of photon energies. X-ray detection is performed si-37 multaneously across a spectrum of energies using dispersive 38 spectrometers employing the Von Hamos design. Different 39 energies of X-rays undergo diffraction at distinct locations on 40 the surface of the crystal analyzer. The X-rays that have un-41 dergone diffraction are directed towards a detector capable of 42 spatially differentiating between X-rays with different energy 43 levels. Scanning instruments utilizing Rowland circle geom-44 etry offer an improved signal-to- noise ratio but necessitate 45 a more intricate mechanical design [13]. To maintain the 46 Rowland condition, both the analyzer crystals and detector 47 are adjusted to change the Bragg angle. Silicon or germa-48 nium is commonly used as materials for the analyzer crystals, with multiple crystal reflections required to cover absorption 50 edges relevant to various elements within the range of X-ray wavelengths. Prior to this work, numerous theoretical calculations on energy resolution were conducted [14]. Therefore, this paper focuses on the introduction and application of a tender energy spectrometer.

This paper introduces the world's first laboratory spectrom-60 eter capable of operating in the tender energy range. The 57 instrument is specifically designed to facilitate research on 58 XAFS within the energy range of 2.0-9.0 keV. The spec-59 trometer design is based on Rowland circle geometry, featur-

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61 spherically bent crystal analyzer, and a silicon drift detector 109 sess a bending radius measuring 500 mm and exhibit a sur-62 (SDD). The paper continues as follows: In Section II, we out- 110 face diameter of 100 mm. To cover as wide a working range 63 line the spectrometer's design and its setup for conducting 111 as possible within the 2.0–9.0 keV energy range and the spec-64 XAFS measurements. Section III details the experimental ar- 112 trometer's angular scanning range (55°-80°), we are gradu-65 rangement employed. Section introduces the performance of 113 ally expanding our collection of crystal analyzers. Currently, 66 the spectrometer. The subsequent section V, encompasses the 114 the available analyzer crystals include: Si (111), Si (220), Si 68 erations that advocate for the extensive utilization and further 116 (620). Table 1 provides an overview of the spectrometer crys-69 advancement of laboratory-based approaches. Finally, in Sec- 117 tals currently accessible, including their respective coverage 70 tion VI, we provide a concise summary.

II. SPECTROMETER DESIGN

The spectrometer is equipped with a laboratory X-ray 73 source, a bent Johann-type spherical crystal monochromators, 74 an SDD, and a vacuum chamber, all devices controlled by 75 LabVIEW. Fig. 1(a) illustrates a schematic diagram of the 76 main components.

A. X-ray source

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Initial X-ray spectroscopy experiments were carried out us-79 ing hermetically sealed X-ray tubes [15]. The development of more powerful X-ray sources for laboratory data-collection systems necessitates addressing the primary concern of effec-82 tively dissipating heat generated by accelerated electrons col-83 liding with the metallic target. Excillum Inc. [16] has devel-84 oped Metal-Jet X-ray tubes, which are conventional micro-85 focus tubes that utilize a liquid metal jet instead of a solid-86 metal anode [15]. The metal-jet supports higher electron 87 beam power, thereby generating higher X-ray flux. There-88 fore, the D2 from Excillum Inc. was selected for laboratory 89 XAFS measurements. The X-ray source is a high-purity liq- $_{90}$ uid gallium jet anode. The tube has a focal spot size of 20 μ m $_{91}$ imes 80 μ m, which transforms to a point source size of 20 μ m imes $_{92}$ 20 μm at a 14.5° take-off angle. The apex angle (opening an-93 gle) of the X-ray cone is approximately 8°, covering a circular 94 area of 70 mm diameter on the crystal. The maximum accel-95 erating potential is 70 kV, and the maximum current is 3.57 ₉₆ mA. The X-ray source window is a 50 μ m thick beryllium 97 window that transmits 2 keV X-rays only half, and almost all 98 (more than 82%) above 3 keV.

B. Crystal

Johann-type Spherically Bent Crystal Analysers (SBCAs) are used for monochromatizing and focusing polychromatic X-ray "bremsstrahlung" energy. In order to minimize the impact of strain fields resulting from spherical bending on 104 energy resolution, crystal wafers are sliced into strips mea-105 suring 15 mm in width prior to being bonded with the glass 141 106 concave substrate. These strips have energy resolution sim- 142 chamber. The crystal analyzer is mounted on a motorized

60 ing a polychromatic micro-focus X-ray source, a Johann-type 108 are purchased from XRS LLC Inc. [17]. The SBCAs pospresentation and analysis of results, accompanied by consid- 115 (311), Si (400), Si (331), Si (422), Si (533), Ge (110), and Ge 118 within the first order of reflection across the working range and theoretical energy Darwin width corresponding to those 120 reflections. There is an energy gap between 2.4-3.2 keV due 121 to the fact that Si or Ge do not have crystal planes suitable 122 for the intermediate energy range. Other crystalline mate-123 rials, such as quartz, can cover this energy range; however, processing quartz spherical crystals is still a challenge.

C. Detector

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The spectrometer is equipped with an Amptek Fast SDD 127 for transmission XAFS measurements, effectively capturing 128 the intensity of the diffraction signal throughout the data collection process. The Amptek Fast SDD is a vacuumcompatible detector with excellent energy resolution that suppresses higher-order harmonics in the diffraction signal while minimizing background noise. It features a 500 μ m thick sil-133 icon sensor layer bonded to the top of the electronic layer, providing an effective area of 50 mm². The SDD can achieve a counting rate of up to 1×10^6 counts/s with a peaking time 136 constant of 0.2 μ s. Its energy resolution is 123 eV at 5.9 keV. 137 Integrated within the SDD is a two-stage Peltier cooling sys-138 tem designed to prevent heat buildup and ensure efficient op-139 eration.

TABLE 1. Compilation of crystals currently accessible, along with their respective 2d-spacings, Darwin width corresponding to the energy broadening of different crystal plane

ergy broadening or different crystal plane.					
2d (nm)	Energy range (keV)	Darwin width (eV)			
0.6271	2.007-2.413	0.23225-0.31464			
0.3840	3.278-3.941	0.19729-0.23914			
0.3275	3.844-4.621	0.11290-0.13513			
0.2716	4.636-5.573	0.12183-0.14527			
0.2492	5.052-6.073	0.07556-0.09003			
0.2217	5.678-6.826	0.08856-0.10546			
0.2000	6.294-7.567	0.15628-0.18357			
0.1789	7.037-8.461	0.12471-0.14535			
0.1656	7.600-9.137	0.03668-0.04367			
	2d (nm) 0.6271 0.3840 0.3275 0.2716 0.2492 0.2217 0.2000 0.1789	2d (nm) Energy range (keV) 0.6271 2.007-2.413 0.3840 3.278-3.941 0.3275 3.844-4.621 0.2716 4.636-5.573 0.2492 5.052-6.073 0.2217 5.678-6.826 0.2000 6.294-7.567 0.1789 7.037-8.461			

D. Motors and movement

The X-ray source remains stationary outside the vacuum 107 ilar to diced-bent crystals and higher efficiency. All crystals 143 module that provides vertical and pitch angular adjustments

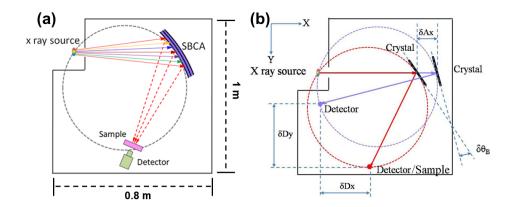


Fig. 1. (a) Design schematic of laboratory X-ray absorption spectrometer based on Rowland circle geometry, (b) Schematic representation of the relative translations (δA_x , δD_y , δD_y , δD_y , $\delta \theta_{bragg}$) of motors A_x , D_y , θ_{bragg} for two different photon energies; the Crystal is maintained on the exact Rowland circle (shown with dotted lines), the center of the coordinate system is the X-ray source.

144 for alignment, as well as horizontal and rotational adjust- 178 148 spectrometer follows the Bragg angle equation and trigono- 182 y-coordinates of the detector. 149 metric formulas, which define the geometric requirements for 150 the Rowland circle.

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The X-ray source, sample, and bent crystal analyzer are 183 $_{152}$ arranged along a Rowland circle with diameter R. With the detector rigidly connected to the sample, which is always directed at the center of the spherical curved crystal by mechanical linkage. For testing, the sample is placed in front of the 156 detector. Whenever there is a change in the Bragg angle θ_B or emitted energy, it becomes necessary to shift the position of the detector along the Rowland circle. Simultaneously, ad-159 justments need to be made to both the angle and distance of the bent crystal analyzer. To maintain optimal focusing geometry, it is essential to keep the detector at an angle Ω that 162 is twice as large as θ_B . When its surface faces towards the X-ray source, this angle Ω becomes zero. Taking the position of the X-ray source serve as the origin of the coordinate system, the X-axis is defined as the direction from the source to the crystal, and the Y-axis is defined as the direction from the source to the detector. Notably, the bent crystal moves exclu-168 sively along the X-axis. The spectrometer's motor positions 195 $(A_x, D_x \text{ and } D_y)$ for a specific photon energy E (measured in 196 170 eV) are given by:

$$heta_B=rcsin\left(rac{12398.42}{2d_{hkl}E}
ight)$$
 $ax=R\sin heta_B$
 $ax=R\sin heta_B$
 $ax=R\sin heta_B\cos^2 heta_B$

 $Dy = 2R\sin^2\theta_B\cos\theta_B$

Where R is the radius of the spherical bent crystal analyzer ments for energy scanning. The goniometer holds the detection (Rowland circle diameter), θ_B is the Bragg angle, d_{hkl} is the the tor, and it is positioned on two linear stages called D_x and two despacing for the given crystal orientation/type used, A_x is 147 D_y in Fig. 1(b). The energy scanning mechanism of the 181 the x-coordinate of the bent crystal, D_x and D_y the x- and

Vacuum chamber

The soft X-ray monochromator employs a custom vacuum the chamber with a pressure of 10^{-6} mbar, achieved using a 700 L/s vacuum pump. To prevent mechanical deformation 187 from pressure differences, all mechanical components are 188 mounted on a separate sturdy steel plate within the vacuum chamber, ensuring the alignment of the diffraction plane. Due to the volume limitations of individual components, achieving proximity during operation proved challenging, resulting 192 in vacuum chamber dimensions of 1 m \times 0.8 m \times 0.6 m and 193 Bragg angles ranging from 55 to 80 degrees.

III. EXPERIMENTAL SETUP

XAFS measurements are conducted in transmission mode. The emission power and spot position of the Metal-Jet X-ray 197 tubes are highly stable, allowing separate measurements of transmitted and direct beams using the same detector. All tests were conducted using the laboratory tender XAFS spec-(1) 200 trometer at the Shanghai Institute of Applied Physics, Chinese Academy of Sciences.

In order to demonstrate the capabilities of the instrument, 203 seven samples are shown in Table 2. Due to the presence of 204 spherical aberration, there is a noticeable difference in the fo-205 cal lengths along the vertical and horizontal directions at the 206 detector side. This discrepancy varies depending on factors 207 such as the Bragg angle and crystal plane. The measurements 208 indicate an approximate range of 4-6 mm for the vertical fo-209 cal length and 1-2 mm for the horizontal focal length. For

(4) 210 XAFS testing in transmission mode, high sample homogene-

211 ity is crucial in both laboratory and synchrotron radiation se-212 tups [18]. By placing a slit in front of the sample, the spot 213 size can be adjusted accordingly. Additionally, a uniform 10 214 mm sample can be obtained by pressing, ensuring uniformity 215 over the spot scale.

The test conditions of the samples are shown in Table 2. All metal foils were purchased from Exafs Materials Inc. [18], and ${\rm TiO_2}$ was purchased from Aladdin Inc. [20]. The beam intensity was measured without the sample (I_0) and with the sample (I_t) , with each scan consisting of 350 energy points, a counting time of 5 s per point, and a motor delay time of 1 s per point. The minimum energy step was set to 0.1 eV. The dead time of all the detectors was less than 25%.

TABLE 2. Summary of sample test conditions.

-	IDLL 2		ar j 01 50	inpro to		101101		248
		Ti K		U M ₅	Th M ₅	Co K	Ni K	249
		edge		edge	edge	edge	edge	250
Absorption		4966		3552	3332	7709	8333	- 251
edge		eV		eV	eV	eV	eV	
Crystal		Si400		Si220	Si220	Si533	Si444	252
analyzer								253
Reflection		1		1	1	1	1	254
(n)								255
Bragg		66.865		65.367	75.681	76.153	71.626	256
angle θ_B								257
samples	rutile		Ti foil	UO_2	ThF_4	Co foil	Ni foil	258
	TiO_2	TiO_2						259
Voltage	21/	21/	21/	30/	30/	30/	40/	260
(kV) /	3.57	3.57	3.57	3.30	3.30	3.30	3.00	261
Current								262
(mA)			_					263
Thickness	-	-	6	-	-	4	6	264
$(\mu \mathrm{m})$					0.40			
monochr-		1.30×1.35			2.10×10^{-5}		2.50×10^{-5}	265
omatic	10^{5}	10^{5}	10^{5}	10^{5}	10^{5}	10^{5}	10^{5}	
flux with								
Sample								
(counts/s)	0.120	0.120	0.120	0.214	0.200	0.027	0.047	266
Energy	0.130	0.130	0.130	0.214	0.200	0.037	0.047	
Darwinian	12	12	12	33	6	1	97	
width (eV)								267

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SPECTROMETER PERFORMANCE

A. Monochromatic Flux

The monochromatic flux was tested with Ge (620) crystals in the energy position of $7040-7050\,\mathrm{eV}$ (Fig. 2(a)). When the voltage was held constant and the current was reduced, the counting rate decreased. The counting rate corresponding to the same power was similar. When the current was held constant and the voltage was increased, the counting rate increased, reaching a maximum at $60\,\mathrm{kV}$, $3.5\,\mathrm{mA}$, and $210\,\mathrm{W}$ ($\sim 5 \times 10^5\,\mathrm{counts/s}$).

B. Energy Resolution

It is difficult to directly evaluate the energy resolution of 236 laboratory source systems; there are two factors contributing 237 to the total energy resolution: the energy broadening corre-238 sponding to the core-hole lifetime and the intrinsic energy 239 resolution of the spectrometer. To characterize the spectrom-240 eter's energy resolution, we used the characteristic peaks of 241 the X-ray source as a sample to provide a strong signal. The 242 X-ray source operates at 120 W (40 kV, 3 mA) and uses a 243 Si553 crystal monochromator. Fig. 2(b) shows the test re-244 sults: peaks A and B are fluorescence peaks for gallium $K\alpha_2$ ²⁴⁵ (9223.8 eV, Bragg angle 71.905°) and $K\alpha_1$ (9250.6 eV, Bragg ²⁴⁶ angle 71.403°). The full width at half maximum (FWHM) of peaks A and B are 2.96 eV and 2.89 eV, respectively. The en-248 ergy broadening corresponding to the core-hole lifetime of the Ga K $lpha_2$ and K $lpha_1$ peaks are 2.66 eV and 2.59 eV, respectively [21]. The final energy resolution of the instrument (ΔE) is 1.298 eV at 9223.8 eV and 1.28 eV at 9250.6 eV.

Additionally, we have theoretically derived the instrument resolution for different energy ranges (Eq. 6). First, we obtained the ΔE_1 of the crystal based on the tested $\Delta \theta$ and the crystal energy at 71.905°. ΔE_1 is the difference between the energy resolution of the spectrometer and the Energy Darwinian Width of the crystal. In the laboratory spectrometer system, different energy ranges correspond to different crystal diffraction planes. The Darwin width correction for each diffraction plane is applied to obtain the final instrument resolution (the Darwin width of each crystal at Bragg angle 71.905° is obtained from XAS X-ray data for the elements). As shown in Table 3, the energy resolution ranges from 0.36 to 1.30 eV at 2.0 to 9.0 keV, covering the instrument's energy range.

$$\frac{\Delta E}{E} = \cot \theta \cdot \Delta \theta \tag{5}$$

where the θ is the Bragg angle, ΔE is the Intrinsic energy resolution and the E is the energy.

TABLE 3. resolution for different energy ranges. (The $\Delta\theta$ is $0.00043\,\mathrm{rad}$, the Bragg angle is 71.403° for Si553 at $9250.6\,\mathrm{eV}$ and 71.905° for others.)

Crystal	Energy (eV)	ΔE_D	ΔE_1	ΔE
Si553	9223.8	0.02707	1.29572	1.296
	9250.6	0.02715	1.28171	1.282
Si422	5882.9	0.08275	0.82640	0.831
Si331	5234.3	0.07062	0.73529	0.739
Si400	4803.3	0.11389	0.67475	0.684
Si311	3982.7	0.10566	0.55947	0.569
Si220	3396.5	0.18496	0.47712	0.511
Si111	2079.9	0.22680	0.27985	0.360

 ΔE_D is Energy Darwin Width, ΔE is Spectrometer Energy Resolution, $\Delta E = \sqrt{\Delta E_1^2 + \Delta E_D^2}$

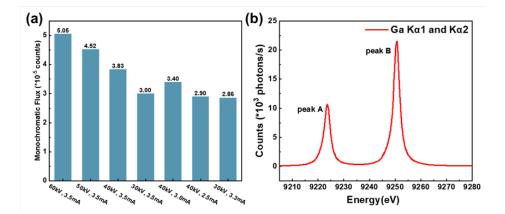


Fig. 2. (a) The monochromatic flux was tested with Ge (620) crystals in the range of 7040-7050 eV in different powder, (b) The fluorescence peaks of gallium $K\alpha_2$ and $K\alpha_1$ were obtained by using the characteristic peaks of the X-ray source.

RESULTS AND DISCUSSION

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A. XANES

In Figs. 3(a) and 3(b), the normalized K-edge X-ray absorption near edge structure (XANES) of titanium foil and anatase TiO₂ are shown alongside synchrotron data from BL14W1 of the Shanghai Synchrotron Radiation Facility (SSRF). All XAS data were background removed and normalized using the software Athena [22]. The Bragg angle, denoted as $\theta = 66.865^{\circ}$, was determined based on the first peak observed in the derivative spectrum of Ti foil and assigned ²⁷⁹ a value of 4966 eV. The consistency of the spectra shown in ₃₁₉ (SSLS). The XAFS data were normalized using the soft-280 Fig. 3(a) suggests that the energy resolution of the laboratory monochromator is similar to that (1.0 eV) achieved by using a double crystal Si(111) monochromator for synchrotron. associated with directly assessing energy resolution in laboratory source systems, encompassing both the measurement of 286 FWHM and comprehensive characterization of the entire en-287 ergy spectrum. The FWHM of the pre-edge peak of the Ti foil 288 is about 1.2 eV. All features can be reproduced, demonstrating that the lab device is suitable for a wide range of applications. 289

290 291 292 sample, while features B and C appeared at the same energy 336 structure modulation among polymetallic sites. as in anatase.

In our previous study, we employed a tender X-ray monochromator to analyze MXene materials (a kind of 2D 337 materials [29]), depicted in Fig. 3(d). Our primary focus was on performing near-edge structure analysis of Ti₃AlC₂ 338 ₃₀₂ and Ti₃C₂Tx materials (a type of MXene), particularly those ₃₃₉ extended X-ray absorption fine structure (EXAFS) measuregos pertinent to supercapacitor applications [23]. Furthermore, 340 ments to extend up to or beyond 1000 eV above the absorp-304 we thoroughly investigated structural attributes and valence 341 tion edge. Ni and Co foils were selected as samples for dif-305 changes, building upon these findings.

Unlike the actinide L-edge, the M-edge of actinide elements shows less core energy level broadening and greater sensitivity to the valence states. However, the M-edges of Th (3.3 keV) and U (3.5 keV) are outside the range of hard X-³¹⁰ rays (> 5 keV) and have not been effectively tested using lab-311 oratory X-ray absorption spectrometers. They are within the 312 energy range suitable for tender X-ray monochromators. The 313 Th and U M-edge absorption spectra were first experimen-314 tally examined using a laboratory light source, as depicted in 315 Fig. 3(e) and (f).

Fig. 3(f) shows normalized XANES spectra of UO₂ and UO₂(NO₃)₂, alongside synchrotron data obtained from 318 beamline XAFCA at the Singapore Synchrotron Light Source 320 ware Athena [22]. Compared with XAFCA, SSLS data, the 321 UO₂(NO₃)₂ and UO₂ spectrum collected by the laboratory 322 spectrometer show a high degree of agreement, indicating that Nevertheless, it is crucial to recognize the inherent challenge 323 the spectrometer's energy resolution is comparable to that of 324 synchrotron radiation near 3.5 keV. All observed features are 325 reproducible, and this level of replication is adequate for most 326 applications.

In the nuclear energy sector's efforts to immobilize ac-328 tinide waste efficiently (Fig. 3(g)), we employed conven-329 tional U-M4 edge XANES spectra to test the valence state of The XANES spectra of TiO₂ in anatase and rutile forms 330 uranium ions and their coordination environment in U-doped can be observed in Fig. 3(c). The spectrum consists of pre- 331 La₂Zr₂O₇-MS samples [24]. In the realm of efficient cataedge components A₁-A₃, a distinctive shoulder B, and pri- 332 lysts (Fig. 3(h)), XANES analysis detected an upward shift in mary peaks C_1 – C_3 . Other reports [26–28] have described the 333 the U-M5 XANES spectra obtained from transmission mode origin of these features. In the rutile TiO₂ sample, features A₁ 334 measurements of UCoO₄ catalysts [25], confirming the presand A2 appeared at lower energies compared to the anatase 335 ence of U 6+ and providing critical evidence of electronic

B. EXAFS

The Bragg angle ranges from 55 to 80 degrees, allowing 342 ferent energy ranges. The XAFS data for these samples are

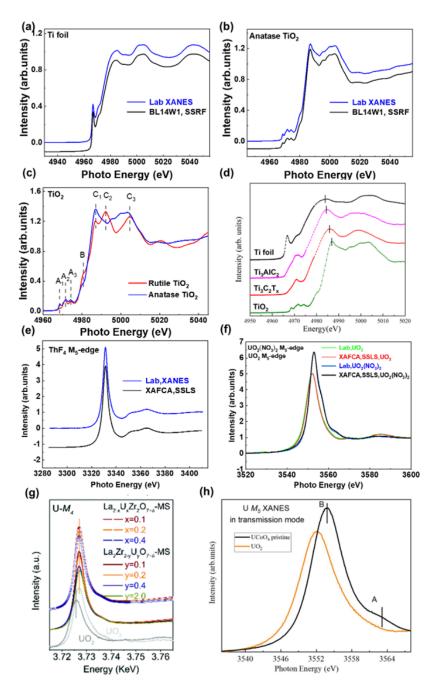


Fig. 3. Normalized K-edge XANES spectrum of (a) titanium foil, (b) anatase TiO2 compared with synchrotron data obtained at SSRF BL14W1; (c) normalized K-edge XANES spectrum of rutile TiO₂ and anatase TiO₂ based on the laboratory spectrometer; (d) Ti K-edge XANES spectra of the pristine Ti₃AlC₂ before and after MS-etching, together with spectra of Ti foil and TiO₂ references. The Ti K-edge XANES patterns of Ti₃AlC₂ (a type of MXene) before and after MS-etching (Reprinted with permission from Ref. [23]. Copyright 2021 Wiley Online Library). Normalized M₅ edge XANES spectrum of (e) ThF₄ and (f) UO₂ compared with synchrotron data obtained at SSLS XAFCA; (g) U-M₄ edge conventional XANES data of U-doped La₂Zr₂O₇-MS samples (Reprinted with permission from Ref. [24]. Copyright 2022 Royal Society of Chemistry); (h) U-M₅ XANES spectra of UCoO₄ (black) and UO₂ (orange) (Reprinted with permission from Ref. [25]. Copyright 2021 ACS Publications).

343 presented in Fig. 4. Figs. 4c and d show the k-space spectra 348 Figs. 4 (a-f). 344 for the two samples, while Figs. 4e and f display the R-space 345 spectra. The EXAFS spectra obtained from the laboratory 346 spectrometer and Beamline 14W1 at the SSRF are compa-

In the realm of electrocatalytic CO₂ reduction (Fig. 4 (g-i)), 350 the transition metal EXAFS of MPc (M=Fe, Co, Ni) catalysts 351 supported on carbon nanotubes were analyzed using the tenrable in their k-space oscillations $(2-12\text{ Å}^{-1})$, as shown in 351 supported on carbon nanotubes were analyzed using the condination number

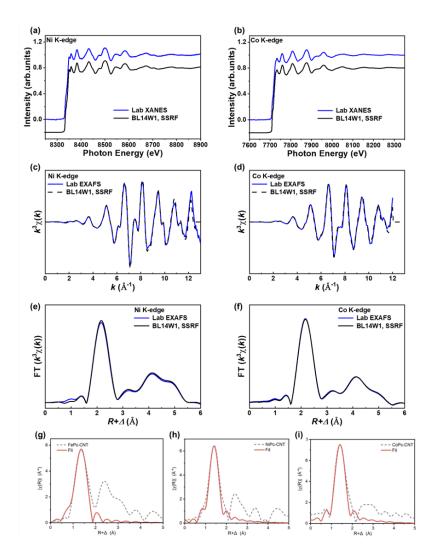


Fig. 4. (a) Ni K-edge XAFS spectrum; (b) Co K-edge XAFS spectrum; (c) Ni K-edge XANES spectrum; (d) Co K-edge EXAFS spectrum; (e) Ni K-edge FT-EXAFS data; (f) Co K-edge FT-EXAFS data; EXAFS spectra of three MPc-CNT samples: (g) FePc-CNT at Fe K-edge; (h) NiPc-CNT at Ni K-edge; and (i) CoPc-CNT at Co K-edge. (Reprinted with permission from Ref. [30]. Copyright 2023 Wiley Online Library)

354 significantly contributing to the elucidation of the catalyst's 360 preliminary characterization of materials prior to synchrotron 355 structure.

VI. CONCLUSIONS AND OUTLOOK

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357 358 laboratory-based XAFS spectrometer utilizing an X-ray 367 similar to other X-ray based experimental methods.

and bond length were ascertained through the fitting results, as source. Laboratory investigations play a crucial role in the analysis. The enhanced configuration incorporates a focus-362 ing mirror and positions the sample behind the X-ray source, thereby enabling a broader energy range due to the utilization of high-power X-ray sources and high-count rate detectors. With accessible laboratory spectrometers, XAS has the poten-We have presented the design and performance of a 366 tial to become a standard method for sample characterization,

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